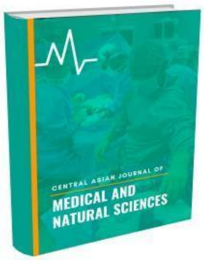

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Article

Synthesis Nanoparticles of MgO as Adsorption Surface for Extraction and Separation of Crystal Violet Dye from Aqueous Solutions

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Abstract: : This study included first the preparation of nano magnesium oxide, which was prepared by sol gel technique, its most important nano-properties were diagnosed and determined by conducting a set of analyses such as X-ray diffraction, FTIR spectra and surface area. After that, we used nano magnesium oxide particles to remove an organic dye in previously prepared aqueous samples, which is crystal violet (CV) dye. The adsorption method was used to remove the dye, the excellent adsorption capability was evaluated in the presence of a set of influential factors such as dye contact time, pH, initial dye concentration, adsorbent dose for MgO, and temperature on crystal violet day. Different values of pH were taken and the best value to obtain an excellent adsorption capacity was pH = 10-12 and the best primary concentration of dye was 400 mg/l and the ideal temperature was 25 ºC while the appropriate dose of the adsorbent was equal adsorbent dose from(0.005 to 0.040 g) contact. The contact time sufficient for adsorption to occur was 120 minutes. In this study, it was proven that the adsorption process was an exothermic process and also the process was largely spontaneous through thermodynamic analysis and the material used to remove the crystal violet dye was excellently effective in aqueous solutions under suitable conditions. It was also found that the Langmuir isostatic model applies excellently to the adsorption process using nano magnesium oxide and that the quasi-second model is the model that the adsorption kinetics follows. Through this, we find that the internal diffusion of the particles plays an important and fundamental role in the adsorption process and thus removes this organic dye from aqueous solutions with very high efficiency.

Keywords: nanoparticles, MgO, crystal violet dye, adsorption, extraction, separation and aqueous solution

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1. Introduction

One of the metal nano-particles that is widely employed in consumer goods is magnesium oxide (MgO), which has a strong adsorption capacity. One of the metal nanoparticles that is widely employed in consumer goods is magnesium oxide, which has a strong adsorption capacity [1]. Nanotechnology has garnered significant attention in science and other technological fields in recent years [2], because of their highly sought-after, distinctive qualities, and range of sizes, compositions, and forms of nanoparticles [3]. The synthesis of nanoparticles can be achieved through a variety of techniques, including sol-gel, co-precipitation, and mechanic chemical methods. Numerous effective devices that are based on nanotechnology have been developed, offering advantages in terms of simplicity, affordability, high purity, large surface area, and excellent physicochemical properties like high stability, optical qualities, tunable porosity, and catalytic activities [4-7]. Nanomaterial's are materials whose size varies between (Monomer, which exhibits 100 - 1 Ben properties) Unique and new, different from the components that were produced with this consideration, it makes them materials that includes in a wide range of aspects of life and technology (biomedical industries such as medical biology) Intents, catalysts, and energy reactions of lac Magnesium oxide is a raw material [8]. As solar of very great importance and widespread in the field nanotechnology; What distinguishes it from its use as a catalyst and ivory [9] For reactions and treatment of toxic silver coatings, superconducting products, and activities. Numerous studies have been conducted on antibacterial agents [10]. Recently, inorganic materials were combined with organic Polymers act as mechanical and thermal improvers [11]. and photo catalysts and antioxidants. A class of substances known as dyes has been utilized since antiquity. Originally, natural plant and animal resources were used to make dyes. The method of gaining was challenging and not very effective. The applied substrate's color fastness was comparatively low, and the shade range was constrained [12,13] . As chemistry advanced, people progressively turned to creating synthetic hues. Nowadays, synthetic dyes are the most commonly utilized because of their extensive color range and greater resistance to the effects of different external agents [14]. The cosmetic, textile, plastic, paint, pharmaceutical, and food sectors employ a vast array of dyes, which eventually results in the presence of these dyes in significant volumes of wastewater. Dye removal from wastewater is crucial due to the hazardous, cancer-causing, and mutagenic nature of these substances [15]. Crystal violet is among the dyes that is used most frequently. It is extensively utilized in the printing ink, paint, and textile industries. It can also be used as a coloring agent, animal medication, or disinfectant in human and veterinary medicine. It is a poisonous, mutagenic, and carcinogenic material that can lead to cyanosis, cancer, respiratory and kidney failure, skin and eye irritation, and elevated heart rate. Even at low quantities, it can prevent aquatic plants from photo-synthesizing and have an impact on aquatic life if it finds its way into natural effluents. It has a strong chemical stability and is not biodegradable. Thus, before the effluent is released, this dye needs to be eliminated [16]. In the context of water treatment, the process by which solid materials, referred to as adsorbents, draw and hold heavy metal ions from an aqueous medium onto their surfaces is referred to as "adsorption of heavy metals." Numerous processes, including complexation, chemisorption, electrostatic interactions, and ion exchange, contribute to this phenomena [16].

2. Materials and Methods

$Mg(NO_3)_2.6H_2O$, sodium hydroxide (NaOH) chloroform solvent, ethanol, and distilled water (all double distal water) from Sigma-Aldrich and BDH. X-Ray, (SEM) Scanning electron microscope visible spectroscopy, UV spectrophotometer, infrared spectroscopy and Ostwald viscometer were used.

A. Adsorption studies

In this research, in order to know and confirm the ability of the adsorbent to removing the dye with high efficiency, the practical part was carried out by dissolving 1 gram of crystal violet dye in distilled water with a known volume in a 1000 ml beaker, after which the solution was shaken vigorously for a period of time of approximately 5 minutes in order to completely dissolve and obtain a homogeneous solution. After that, it is possible to take the other different concentrations using the known dilution methods in a serial manner. The effect of some important factors affecting the adsorption process was observed based on the well-known evaporative adsorption method. Examples of these important factors are pH, initial dye concentration, contact time, adsorbent dose and temperature. When checking the effect of pH, 25 ml of organic dye solution was added, then 25 0.0 of nano magnesium oxide was added in a flask where the pH of the dye was fixed between 2 - 10. The pH was controlled using dilute hydrochloric acid and sodium hydroxide at concentrations of 0.1 M and was controlled by a pH meter (ATC Instrument, digital model). For the adsorption experiments, 250 ml of crystal violet solution was mixed with 0.25 g of adsorbent in a conical flask and placed in a shaker (Julabo , Germany, rpm=10-200). Samples were taken at predetermined intervals (120 minutes), and the crystal violet solution separated from the adsorbent using a micro-pipette.

Dose of MgO was varied between (0.005 and 0.04) g in 25ml of crystal violet solution to find the optimal amount of adsorbent. The effect of dye concentration was studied by preparing 25 ml of dye solution in a conical flask with concentrations ranging from 25-400 mg/L for crystal violet dye, using an MgO dose of 0.025g/L, and placing the flasks in a shaker. Maintaining the temperature at 25 °C. A spectro-photometer (TRSP-722 MD-1105 UV-Visible Spectrophotometer) was used to measure absorbance, and the temperature effect was experiments at 298, 308, 318 and 328K with a shaker rate of 250 RPM.

The amount of dye removed by the sorbents (q_e) and the percentage of dye extracted (%E), it was calculation of extraction by :

$$\%Extracted = (C_o - C_e) / C_o \times 100 \qquad (1)$$

$$q_e = (C_0 - C_e) * V / m \qquad (2)$$

The variable qe represents the amount of crystal violet adsorbed (in mg/g). C0 and Ce refer to the initial and equilibrium concentrations of crystal violet in the solution phase (in mg/L), respectively. V denotes the volume of the solution, while m indicates the mass of the MgO used (in grams).

B. Preparation of nan-magnesium oxide (MgO)

The preparation of magnesium nitrate involved applying 0.2 mol/L of sodium hydroxide (NaOH) to 250 ml of distilled H_2O, stirring continuously for 45 minutes, and mixing 1 M of NaOH with distilled water in a large-capacity beaker (250 ml). After 45 minutes of continuous stirring, the sodium hydroxide was added drop by drop to the magnesium salt in the reaction flask, which had a 500 ml capacity, and thoroughly mixed with a magnetic stirrer. After two hours at ambient temperature, the product was in the form of a milky precipitate. It was filtered and repeatedly cleaned with distilled water before being dried in an oven where it burned for two hours at 300°C [11].

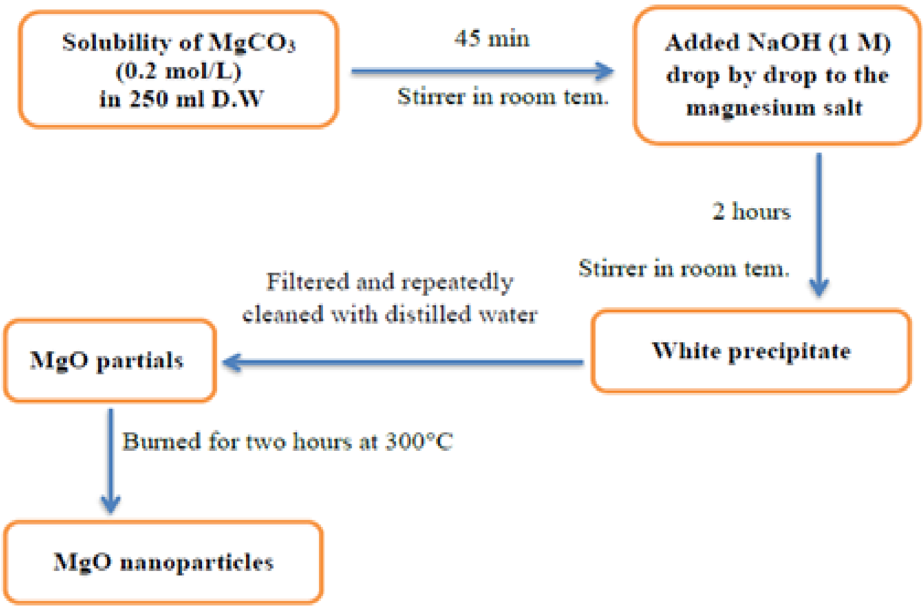

Figure 1. MgO nanoparticles preparation.

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3. Results

A. Characterization of prepared adsorbent

1. MgO nanoparticle physio-chemical characterization

Using a UV-Vis Spectrophotometer, the samples' surface Plasmon resonance was investigated at a resolution of (1 nm) between (250 -800 nm). SEM imaging was used to characterize the morphology of the nanoparticles, and an XRD research was conducted to ascertain their shape. The crude extracts' infrared spectra were acquired using an FT-IR Spectrophotometer, and they ranged between (400 - 4000) cm-1. The presence of functional groups in the produced sample was ascertained by analyzing the stretching frequencies and symmetric and asymmetric stretching [17].

2. X-ray diffraction

X-ray diffraction patterns of the prepared nano-magnesium oxide were compared with the reference standard JCPDS No. 5910-000. (98). The diffraction peaks (100, 111, 200, 220, 222, and 311). Are located at angles (78.83o, 74.8o, 62.25o, 43o, 37o and 34.3o, respectively) as shown figure (1), which indicate and confirm that the nano-magnesium oxide powder is polycrystalline and has a face-centered cubic structure (fcc) according to the reference standard. When it was associated with the JCPDS (Join committee on power diffraction standards), it was found that it is identical and its structure is (fcc) and is consistent with the results of the literature [18] .

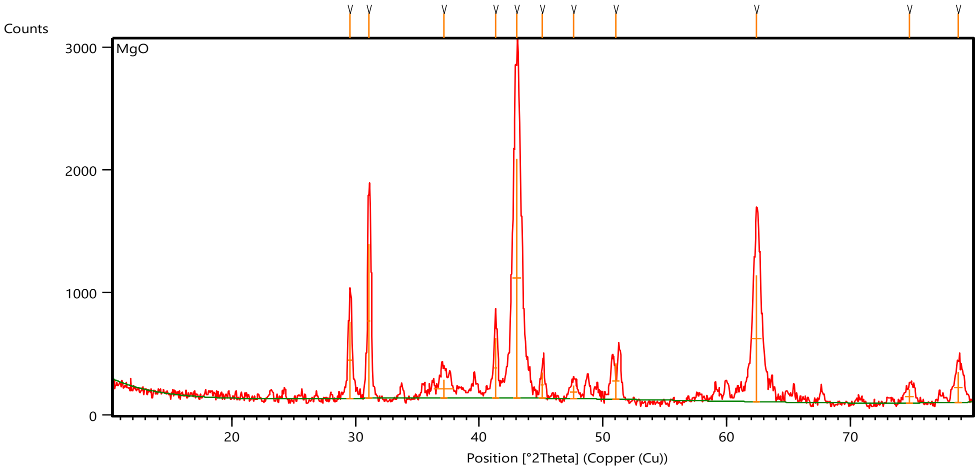

Figure 2. X-ray diffraction of MgO.

3. FTIR spectra

We were able to characterize the characteristic bands of the sorbent produced by infrared analysis after immersion in water and continuous air flow. Figure (2) shows the FTIR spectra of the prepare MgO in the range of 400 - 4000 cm-1. It is clear that the produced sorbent shows absorption bands associated with CO2 at 2523 cm-1and 877 cm-1. Moreover, the carbonate group is responsible for the absorption band at 1441 cm-1and 876 cm-1. The OH stretching of surface hydroxyl groups, connecting hydroxyl groups, and adsorbed water molecules is responsible for the appearance of the smooth band at 3413 cm-1 [19, 20].

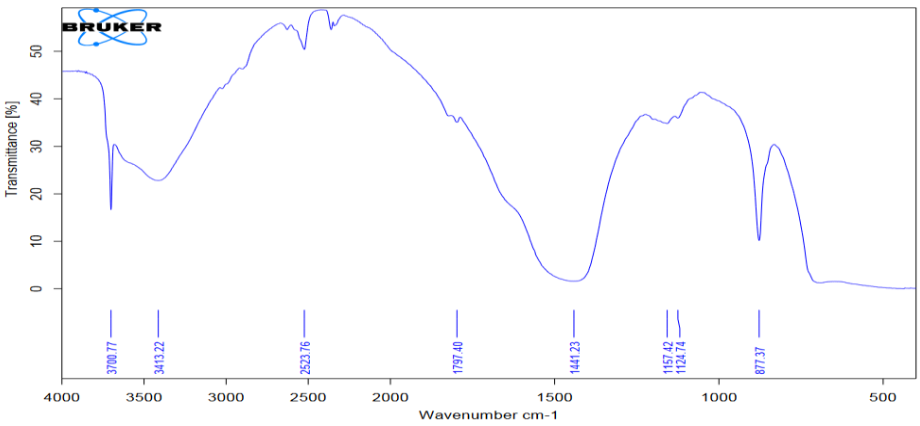

Figure 3. FTIR spectra of MgO after adsorption.

4. Surface area of MgO

Several techniques were the surface area of the prepared sorbent was calculated, and the results are presented and summarized in the figure (3).

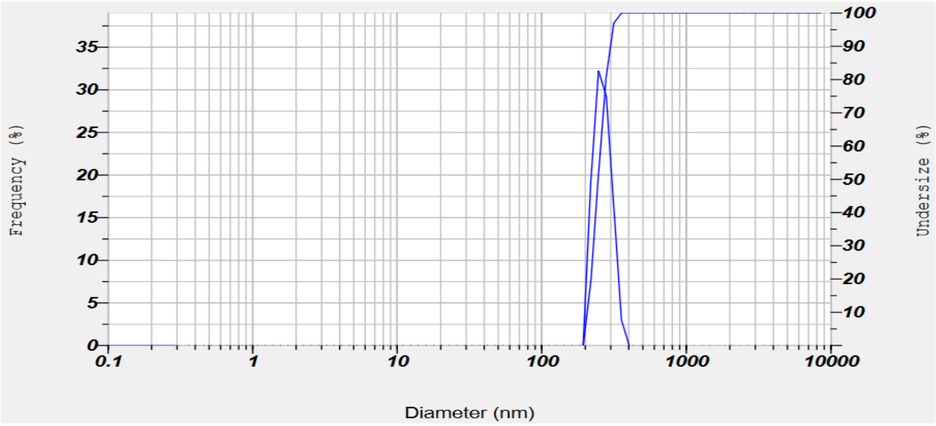

Figure 4. Surface area of MgO.

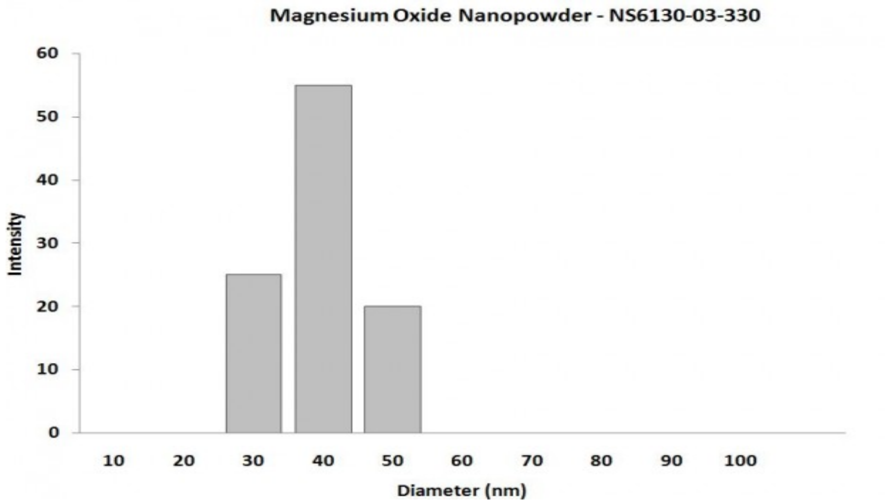

Figure 5. Particles size analysis of MgO [21].

5. SEM (Scanning Electron Microscopy)

The scanning electron microscopy of MgO, done through cryogenic grinding of used tires, reveals its porous structure at magnifications of 50000x and 3200x as shown in figure

(5). The prominent presence of MgO, along with its carbonaceous nature, contributes to the observed porosity in the (SEM) images.

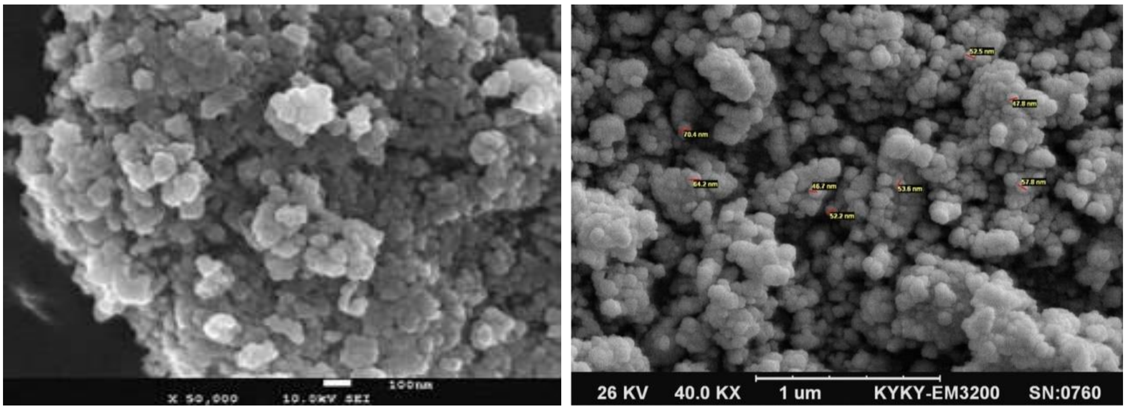

Figure 6. Image SEM spectra for MgO about 50000x and 3200x [21], [22].

B. Study the effect of some factors affecting adsorption

1. Effect of pH on crystal violet dye absorption

Investigating how pH affects the MgO solid phases' ability to remove crystal violet. The results are also shown in figure (6), where the addition of 0.1 M HCI and 0.1 M NaOH adjusts the pH of the solution. The curve increases with increasing pH (2-12) until stability is reached. The study shows that at starting pH = 10, the adsorption of crystal violet dye solution is maximum. However, because the dye began to decompose and change color when the dye solution was between 10 and 12, and because the adsorption process curve is stable at pH values between 10 and 12, the adsorption capacity does not change significantly pH = (10 - 12) [23]. An important factor that affects how the crystal violet dye absorbs onto magnesium oxide (MgO) is the pH of the solution. The process can be affected by pH depending on the surface charge of MgO under low pH and acidic conditions. Because to the protonation of surface OH groups, the surface of MgO may become positively charged at low pH values of 2-6. The positively charged surface of MgO and the cationic crystal violet dye may experience electrostatic repulsion, which may reduce the adsorption effectiveness [24]. Deprotonation of surface hydroxyl groups on magnesium oxide (MgO) may increase the surface negative charge. The positively charged crystal violet dye may be drawn to the negatively charged surface, accelerating the adsorption process. The pH of the solution can influence the ionization form of the crystal violet dye. In general, crystal violet maintains its positive charge across a wide pH range. However, extreme pH levels may modify the dye structure and charge distribution, which may change how it interacts with the adsorbent [25].

Absorption of cationic dyes, such as crystal violet, is generally more effective in an alkaline environment. Research often shows that as the pH approaches alkaline levels, the ability of MgO to absorb crystal violet increases, reaching an optimum level where absorption is maximum. This is mostly because the surface of MgO has a more negative charge, There is an attraction between the sorbent and the dye, which is an electrostatic attraction [26].

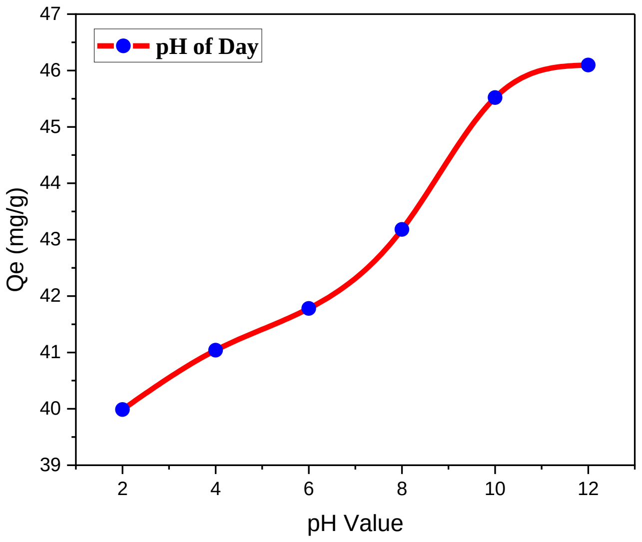

Figure 7. PH values effect on adsorption capacity of CV dye by MgO.

2. Study the effect initial concentration on the dye

The surface area of the MgO sample under study showed adsorption of CV Dye at varying starting concentrations ranging from 25 to 400 mg/l at a At a temperature of 25°C, an analysis of the data presented in the figure was conducted (7), reveals a relationship between the concentration of the dye and the number of binding sites available on the surface of the adsorbent. The relationship is most evident at the beginning of the adsorption process, when the dye concentration is highest and decreases as available sites are occupied. As the dye concentration increases, less of the original concentration is removed. At equilibrium the amount of adsorbed crystal violet increases (13.78 - 345.45) mg/g of adsorbent [27]. At lower concentrations 25 ppm there are fewer dye molecules in the solution with percent 100%. The available surface sites on MgO are more than sufficient to adsorb the dye, leading to high adsorption efficiency. Most of the dye molecules are adsorbed onto the MgO surface, resulting in a higher percentage removal of the dye from the solution. The dye increases at 400 ppm, the number of dye molecules in the solution becomes significantly higher. The available active sites on the MgO surface might become saturated, leading to a decrease in the percentage removal of dye [28]. At lower concentrations 25 ppm the removal efficiency (percentage of dye removed from the solution) is typically high because the number of dye molecules is much lower than the adsorption capacity of MgO. At higher concentrations 400 ppm the removal efficiency may decrease as the MgO becomes saturated with dye molecules. Although the total volume of dye adsorbed increasing, the proportion of dye removed from the solution compared to the initial concentration decreases [29].

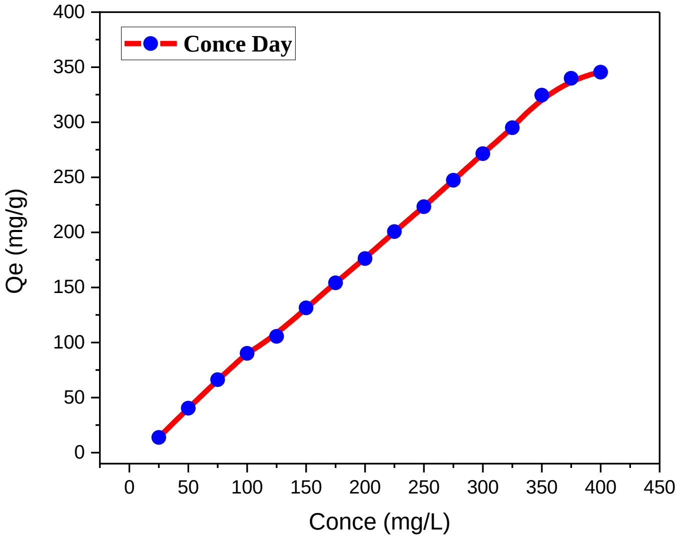

Figure 8. Concentration effect on adsorption capacity of CV dye by MgO.

3. Study of the effect contact time on crystal violet dye adsorption

Crystal violet dye adsorption assay was performed with different contact times (10-120 min) and concentrations according to the sample MgO (250 mg/L). The data shown in Figure 8 show that as the Contact Time increases, a sum total of (CV) absorbed increases until it achieves equilibrium within 25 minutes for the MgO sample. Because the sawdust has a larger surface area accessible to initially absorb the dye, the dye removal rate is higher. The availability of many external sites already occupied and the slow rate at which methyl bromide molecules diffuse into the pores of the different sorbents may be reasons for the slow progress of the second stage. Effect of reaction duration on MgO adsorption capacity of crystal violet dye. It is obvious that as the contact time increased from 0 - 45 min, the adsorption capacity of the MgO adsorbent increased rapidly, reaching most than 70 % of the equilibrium adsorption efficiency of crystal violet dye in only 10 minutes. After that, the adsorption rate was gradually decreased until the adsorption efficiency became constant and the adsorption rate achieved equilibrium, which occurred after 120. Therefore, the contact time for CV dye adsorption onto the sorbents was determined to be 120 min based on the available experimental data. The early stage of rapid adsorption may be due to the increased driving force that facilitates the methyl ions to quickly reach the surface of the adsorbed particles, as well as the exposed availability of active sites in addition to surface area remaining on the adsorbent [30].

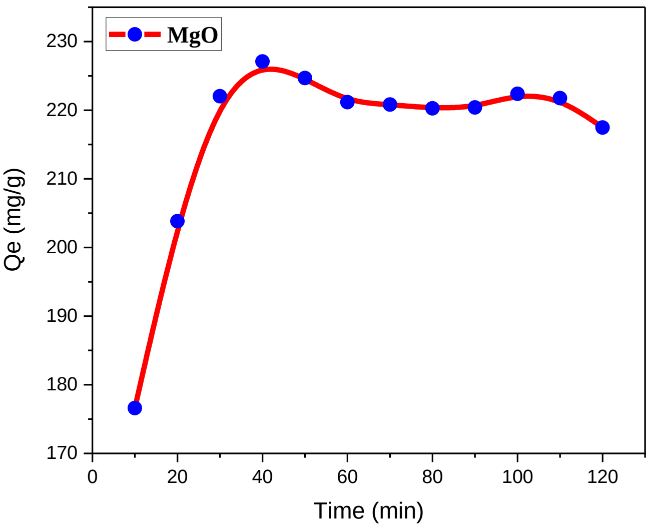

Figure 9. Contact time effect on adsorption capacity of MgO adsorbent.

4. Study of the effect adsorbent dosage for MgO

The percentage of crystal violet dye extracted (%) by MgO increased from (84.45 - 96.11) % as a result of the mass effect on the adsorption of the amount of dye by MgO when the dose of the absorbent material was increased from 0.005 to 0.04 g. The crystal violet dye extraction results in figure (9) show that the dye absorption increases with the adsorbent dose. This is likely due to the high absorption loading of the experiment, which becomes important when transferring the high dye mass from the bulk liquid to the solid surface resistivity [31]. The number of available adsorption sites overgrown with growth adsorption dose, The adsorption sites stay unoccupied throughout the adsorption process. A higher concentration of MgO provides more active sites for the adsorption of CV dye particles. The higher dye removal from solution results from the increased adsorption surface area resulting from increased MgO concentration. Since there are more adsorption sites at larger doses, the adsorption process may approach equilibrium more quickly. At the beginning of the adsorption process, the MgO surface contains a large number of accessible active sites. Due to the rapid absorption of crystal violet dye particles, the absorption rate increases dramatically within the first few minutes. The large unlike in initial concentration beside the dye molecules in the solution and the adsorbed surface results in rapid adsorption of the dye molecules on MgO. As the adsorption process progresses, fewer accessible active sites on the MgO surface become available, causing the adsorption rate to slow down. There are now fewer sites left for the dye molecules to compete for, reducing the adsorption rate. The adsorption process may become limited by the rate of diffusion of dye molecules into the MgO pores or through the boundary layer surrounding the molecules. This can result in a slower overall absorption rate as the process continues [32].

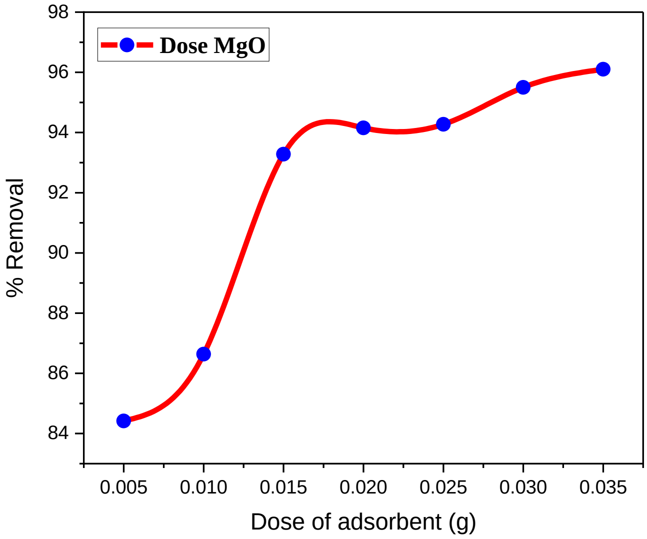

Figure 10. Adsorbent dose effect on adsorption capacity of MgO adsorbent.

5. Study of the effect temperature on removal crystal violet color

For different dye concentrations (100-300 mg/L) per dye shown in figure (10), the impact of temperature on adsorption of crystal violet 0.025g at in 25ml of adsorbent, contact time 180 minute, pH = 8. The stirring speed was checked at 140 rpm. The system temperature increased from 25 to 55 °C, which resulted in an increase in the absorption capacity of MgO. For all the concentrations studied, it was observed that the equilibrium adsorption increases with rising temperature. As the temperature increases, the viscosity of the solution decreases, which enhances the diffusion rate of adsorbed particles through the outer boundary layer and into the inner pores of the adsorbent [33]. The highest percentages of crystal violet removed by MgO at various temperatures were found to be (70.04%-93.39%) at 25 °C, (72.77%-94.81%) at 35 °C, (77 - 83.13 % - 95.71 %) at 45°C, and (79.19-97.41%) at 55°C. This occurrence indicates that endo-thermic adsorption has occurred. This may be caused by increased molecular mobility, which usually increases with temperature [34]. Depending on whether the process is endo-thermic or exo-thermic, temperature has an effect on adsorption. Raising the temperature will enhance the adsorption capacity if the adsorption of crystal violet on MgO is endothermic, as higher temperatures provide more energy to overcome the activation energy barriers of adsorption. On the other hand, if the process is exothermic, raising the temperature can lead to a decrease in adsorption capacity because higher temperatures tend to favor adsorption into the system. The adsorption capacity often increases with temperature because the adsorption of dyes such as crystal violet onto solid sorbents often tends to be endothermic.

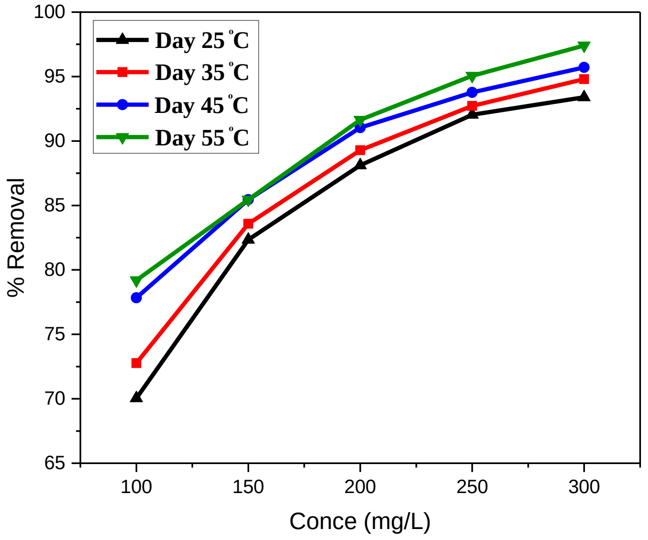

Figure 11. Temperature effect on adsorption efficiency of CV dye by MgO.

4. Discussion

A. Isotherms of adsorption

The experimental adsorption equilibrium data were evaluated using the Freundlich and Langmuir isotherm models, which are calculated as outlined below:

Freundlich isotherm: $\ln q_e = \ln K_f + 1/n \ \ln C_e$ (3)

Langmuir isotherm: $C_e/q_e = 1 / Q\,b + C_e / Q$ (4)

Figures (11 and 12) present Langmuir and Freundlich isotherms for crystal violet dye adsorption onto MgO at various initial concentrations, while tables (1, 2) summarize the constants and correlation coefficients for both models [32].

$RL = 1/ (1+b\ C_0)$ (5)

Table 1. Langmuir isotherm Parameters for CV dye adsorption with MgO

Adsorbents	Parameters of Langmuir			
	R2	Q max, fitted	b (mg/L)	RL
MgO	0.1635	- 719.424	- 0.00021	0.978683

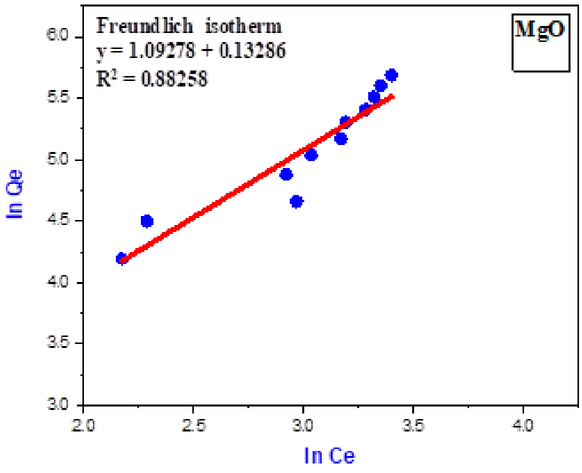

Figure 11. Freundlich plot of adsorption.

Table 2. Freundlich isotherm Parameters for CV dye adsorption with MgO.

Adsorbents	Freundlich parameters		
	R2	K f	l/n
MgO	0.88258	1.79761	1.09278

B. Adsorption kinetics

To explore the mechanism of the adsorption process, including mass transfer and chemical reaction steps, kinetic models can be utilized to analyze the experimental data. The adsorption kinetics of crystal violet dye on modified MgO were assessed using First Order, Second Order, and intra-particle diffusion models. The pseudo-first-order kinetic model can be expressed as follows:

$$\log (q_e - q_t) = \log q_e - (K_1 * t) / 2.303 \qquad (6)$$

As shown in figure 13, based on an concentration CV dye of 250 ppm. The low correlation coefficient (R^2) values and the mismatch beside the particle part and calculated q_e values, derived from the linear curves table (3), this suggests that the adsorption of crystal violet dye onto MgO does not follow a first-order kinetic model. Instead, the process aligns with a second-order kinetic model, as shown by the following equation [32]:

$$t / qt = 1/(K_2 * q_e^2) + t / q_e \qquad (7)$$

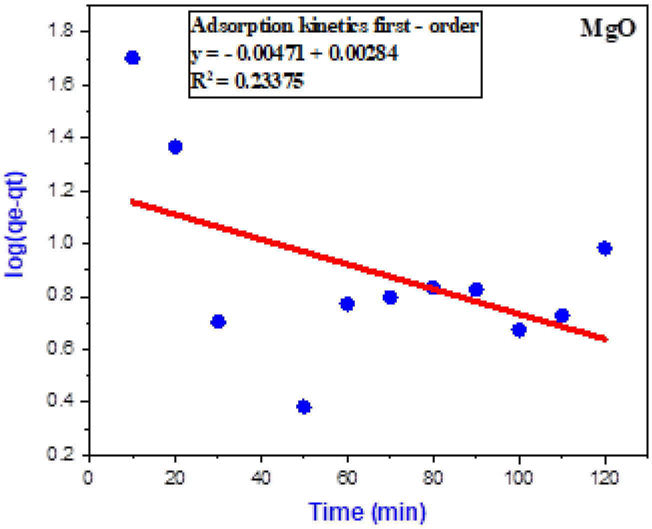

Figure 12. First-order kinetic model.

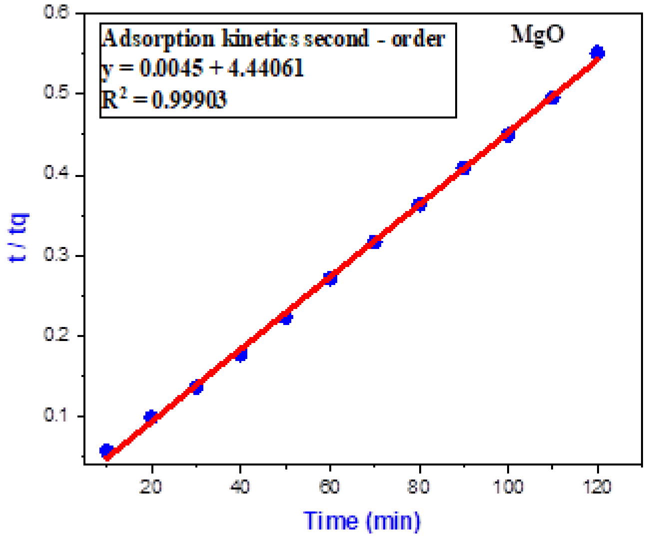

Figure 13. Second-order kinetic model.

Table 3. First -order kinetic for adsorption of of CV bye by MgO.

Adsorbent	First order		
	$q_{e\ cal}$ mg/g	K_1 (min^{-1})	R^2
MgO	- 212.314	- 0.01085	0.23375

Table 4. Second order of kinetic for adsorption of CV dye by MgO.

Adsorbent	Second-order		
	$q_{e\ cal.}$ $mg.g^{-1}$	K_2 $g.\ mol^{-1}.min^{-1}$	R^2
MgO	222.222	0.9753	0.999

C. Thermodynamic parameters evaluation

The temperature dependency of adsorption is related to various thermodynamic parameters. To investigate the thermo-dynamics of the adsorption process, experiments were conducted at 25, 35, 45, and 55 °C. The standard energy change (ΔG^0) was calculated as follows:

$$\Delta G^\circ = \Delta H^\circ + T\,\Delta S^\circ \qquad (8)$$

The values of Δ H0 and Δ S0 were determined using the van't hoff equation:

$$\ln Kd = \Delta S^\circ / R - \Delta H^\circ / RT \qquad (9)$$

The change in enthalpy ΔH^o is associated with the chemical interactions during the process. The negative value of ΔH^o observed in this study confirms that the adsorption process is exothermic [34].

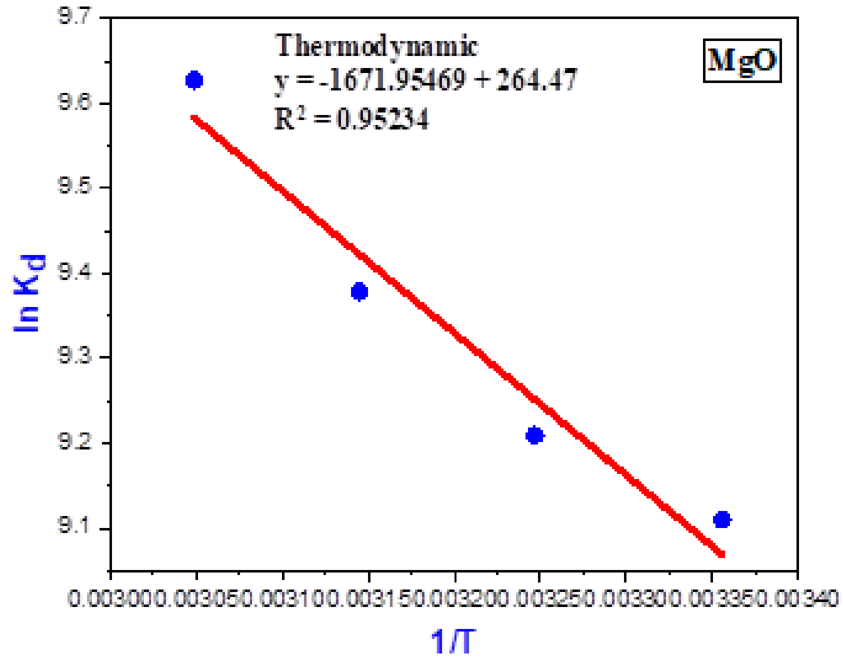

Figure 14. Thermo-dynamic parameters of the adsorption process of CV dye on 250 ppm of MgO.

Table 5. Thermo-dynamic parameters for adsorption of CV dye on MgO at four temperatures.

Adsorbents	**Crystal Violet Concentration (mg/L)**	**ΔHº (KJ/mol)**	**ΔSº (KJ/mol)**	**ΔGº (KJ/mole)**			
				298 K	**308 K**	**318 K**	**328 K**
MgO	250	-13.9006	0.122049	-50.27	-51.49	-52.71	-53.93

5. Conclusion

In this research, nanoparticles of Magnesium oxide was synthesized using a sol-gel chemical method. It can be used to remove crystal violet dye. It showed a high ability to get rid of this dye, as the adsorption process was highly efficient in adsorption of crystal violet dye on magnesium oxide nanoparticles under suitable conditions of a group of influencing factors that can be controlled. The method was easy, fast, low cost and can be easily applied.

The adsorption capacity depends on several factors, including, concentration, pH, temperature, The amount of adsorbent used and the duration of contact time. The maximum removing capacity of crystal violet dye was achieved at 92.05 % for MgO at around 25°C. This was reached at 60 minutes for MgO. The effect of pH , tested within a range of (2 - 12), showed that the maximum absorbing of CV dye on the MgO at pH 8The equilibrium adsorption isotherms were evaluated using the Langmuir and Freundlich models to quantitatively describe the dye adsorption process. The First and Second order models were the models followed by the adsorption process and the internal diffusion carried out by the particles The adsorption process of the crystal violet (CV) dye played a crucial role in the attachment of dye molecules to the surface of the adsorbent material, with the process being both exothermic and spontaneous.

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